## Report Instructions for Migratory Insertion Laboratory

This write-up consists of:

1. Analysis of your spectral data with the purpose of determining the geometry & symmetry of the starting material and two products that you prepared
2. Description of your experimental procedures with your data arranged in a tabular format
3. Annotated spectra. You are encouraged to search for additional references to aid in assignments, but be sure to cite all of these sources appropriately in your report.

**Analysis**

Use the number of CO stretches and the number & type of resonances in the 1H & 13C NMR spectra to propose three-dimensional structures & symmetries of [*η*5‑CpMo(CO)3)]2, *η*5-CpMo(CO)3Me and *η*5-CpMo(CO)2(acetyl)PR3. Even if you know the structures by making reference to other sources, you need to convince your instructors that you understand how your data support your structure assignments. Stronger arguments are made when comparing with alternative structural possibilities. When discussing specific results in the text, refer to the table or figure in which they are located (i.e.: Table I or Figure I). The table or figure should have a title (i.e.: Table I. 1H NMR Data for *η*5-CpMo(CO)3Me).

**Experimental Section**

You should search for references in a good inorganic chemistry such as "Inorganic Chemistry" as models for writing this section. This section will comprise a “General” section, descriptions of the syntheses of each of your two products, and tables of spectral data.

The General section indicates the equipment and the type of instruments used to synthesize and characterize your products. The starting materials MeI, PR3, the 1-M solution of LiEt3BH, and the anhydrous solvents were purchased from Aldrich or Strem and used as received. The [*η*5-CpMo(CO)3)]2 was purchased from Strem and used as received. The alumina (neutral) was purchased from Aldrich and dried at 250 °C under vacuum for 4 hours before use. The deuterated NMR solvents were purchased from Cambridge Isotope Laboratories, degassed, and filtered through neutral alumina before use. You used a solution IR cell with KBr windows for IR data (in CH2Cl2). You should also describe the IR and NMR instruments employed and the frequencies of the different nuclei (you can find them listed as sfrq in the parameters for your spectra from the NMR instrument). For example, the proton frequency for our instrument is 400 MHz (you can look up the 13C and 31P frequencies). Both 13C and 31P spectra were acquired with proton decoupling. Except for 31P, the chemical shifts should be referenced against solvents—for C6D6 the residual proton is at 7.16 ppm and the 13C is at 128.39 ppm. For CDCl3, the residual proton is at 7.24 and the 13C is at 77 ppm. We referenced the 31P NMR spectrum to 85% phosphoric acid (H3PO4, which comes at 0 ppm).

The General section should be followed by descriptions of the individual syntheses performed. You should make clear what activities were performed in an inert-atmosphere glove box. Make sure to specify the quantities (mass or volume/concentration) and mmol or mol used for each reagent. Solvent quantities can be estimates (and so should be rounded off to the nearest 5 mL). At the end each description, report your yield (in actual grams and percentage).

These sections are often followed by lists of spectral data—instead, you will put the data into one or more tables, similar to what you have done for previous lab write-ups in this class. Make sure to specify the solvents used in each spectrum you acquired (probably easiest to provide as footnotes to the relevant tables). Always keep in mind significant figures! For 1H NMR data, chemical shifts are generally reliable to two decimal places and coupling constants to the tenth Hz; for 13C NMR data, the chemical shift is accurate to about one decimal place and 13C–31P coupling constants to the nearest Hz. Assume that 31P chemical shifts are accurate to the first decimal place in their chemical shift value. IR data are accurate only to the wave number integer value (i.e., no decimal places).

**Annotated Spectra**

1H NMR Spectra: Draw your molecule on a copy of your spectrum, assign letters to the chemically distinct protons, and label the peaks in the 1H NMR spectrum with the appropriate letters. Also, make sure the acquisition parameters are displayed on your spectrum.

13C NMR spectra: Draw your molecule on a copy of your spectrum, assign letters to the chemically distinct carbons, and label the peaks in the 13C NMR spectrum with the appropriate letters. Also, make sure the acquisition parameters are displayed on your spectrum.

31P NMR spectrum: Include a copy of this spectrum with the structure of your product drawn. Attempt to assign any impurities. Also, make sure the acquisition parameters are displayed on your spectrum.

IR Assignments: (Do this for both products and the [*η*5-CpMo(CO)3)]2 starting material.) For each peak you are able to assign, indicate the vibrational mode giving rise to

that peak. Expansions of interesting features should be included where applicable. By no

means does every peak in the fingerprint region need be assigned. Make sure that you

identify which peaks are due to solvent (if any).